इंटरनेट मानक

Disclosure to Promote the Right To Information

Whereas the Parliament of India has set out to provide a practical regime of right to information for citizens to secure access to information under the control of public authorities, in order to promote transparency and accountability in the working of every public authority, and whereas the attached publication of the Bureau of Indian Standards is of particular interest to the public, particularly disadvantaged communities and those engaged in the pursuit of education and knowledge, the attached public safety standard is made available to promote the timely dissemination of this information in an accurate manner to the public.

"जानने का अधिकार, जीने का अधिकार"
Mazdoor Kisan Shakti Sangathan
"The Right to Information, The Right to Live"

"पुराने को छोड नये के तरफ"
Jawaharlal Nehru
"Step Out From the Old to the New"

IS 3470 (2002): Hexane, Food Grade [PCD 3: Petroleum, Lubricants and their Related Products]

"ज्ञान से एक नये भारत का निर्माण"
Satyanarayan Gangaram Pitroda
"Invent a New India Using Knowledge"

Act Number 22 of 2005
The Indian Parliament
RIGHT TO INFORMATION

Made Available By
Public.Resource.Org

"ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है"
Bhartṛhari—Nītiśatakam
"Knowledge is such a treasure which cannot be stolen"

BLANK PAGE

BRUTUM FULMEN

IGNORANTIA JURIS NON EXCUSAT

PROTECTED BY COPYRIGHT

IS 3470 : 2002

भारतीय मानक

हेक्सेन, खाद्य श्रेणी — विशिष्टि

(पहला पुनरीक्षण)

Indian Standard

HEXANE, FOOD GRADE — SPECIFICATION

(First Revision)

ICS 75.080

© BIS 2002

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

December 2002 **Price Group 7**

Petroleum Products Sectional Committee, PCD 3

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Petroleum Products Sectional Committee had been approved by Petroleum, Coal and Related Products Division Council.

Formulation of this standard was taken up at the instance of the Ministry of Health, Government of India, for covering the food grade material suitable for solvent extraction of oleaginous materials, particularly to enable the production of solvent extracted vegetable oils. This standard was first published in 1966. Revision of the standard has been undertaken to align it with latest WHO specifications for Food Grade Hexane prepared at the 51st Joint (FAO/WHO) Expert Committee on Food Additives (JECFA) (1998) and published in Food and Nutrition Paper (FNP) 52 Add 6 (1998).

In this revision some new characteristics namely, refractive index, benzene content, polyaromatic hydrocarbon have been included. New test methods for determination of sulphur, polyaromatic hydrocarbon and benzene have been incorporated. Requirements of non-volatile residue, sulphur and lead have been further tightened. Characteristics such as colour, copper strip corrosion, doctor test, phosphorous, chloride and bromine number have been dropped, in view of new stringent specification requirement.

In the preparation of this standard considerable assistance has been derived from Compendium of Food Additive Specifications of Food and Nutrition Paper 52 Add 6 (1998) on hexanes, prepared by joint FAO/WHO expert committee on food additive.

The composition of the Committee responsible for formulation of this standard is given in Annex G.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2:1960 'Rules for rounding off numerical value (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

AMENDMENT NO. 1 JULY 2004
TO
IS 3470 : 2002 HEXANE, FOOD GRADE — SPECIFICATION

(First Revision)

[*Page* 2, *Table* 1, *Sl No.* (ii), *col* 5] — Substitute ‘[P :]1)’ *for* ‘[P : 18]’.

[*Page* 2, *Table* 1, *Sl No.* (iii), *col* 5, *against ‘Distillation’*] — Insert ‘[P : 18]’.

(PCD 3)

Reprography Unit, BIS, New Delhi, India

Indian Standard
HEXANE, FOOD GRADE — SPECIFICATION
(First Revision)

1 SCOPE

This standard prescribes the requirements and methods of sampling and test for hexane, food grade, to be used as the solvent for extraction of oleaginous materials.

2 REFERENCES

The following Indian Standards contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication the editions indicated were valid. All standards are subjected to revision, and parties to agreements based on the standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
170 : 1986	Acetone (*third revision*)
265 : 1993	Hydrochloric acid (*fourth revision*)
1070 : 1992	Reagent grade water (*third revision*)
1260 (Part 1) : 1973	Pictorial marking for handling and labelling of goods : Part 1 Dangerous goods (*first revision*)
1447 (Part 1) : 2000	Petroleum and its products — Methods of sampling : Part 1 Manual sampling (*first revision*)
1448	Methods of test for petroleum and its products
[P:18] : 1991	Distillation of petroleum products (*second revision*)
[P:32] : 1992	Density and relative density (*second revision*)
2631 : 1976	*iso*-Propyl alcohol (*first revision*)

3 REQUIREMENTS

3.1 General

The material shall be a petroleum distillate fraction that contains a high proportion of *n*-hexane and shall be clear colourless liquid with characteristic petroleum like odour, free from sediment, suspended matter and moisture.

3.2 The material shall also comply with the requirements prescribed in Table 1, when tested according to the appropriate methods given in col 4 and 5 of the Table 1.

4 PACKING AND MARKING

4.1 Packing

The material shall be packed and sealed in suitable containers, as agreed to between the purchaser and the supplier and subject to the provisions of the latest issue of *Red Tariff Rules* and Rules for the conveyance by Rail of Explosives and other Dangerous goods, issued by the Indian Railways Conference Association.

4.2 Marking

4.2.1 Each container shall be marked with caution label 'FLAMMABLE' together with the corresponding symbol for labelling dangerous goods, as given in Fig. 3 of IS 1260 (Part 1).

4.2.2 When the material is packed in drums or other smaller containers, it shall also be marked with the following information:

a) Name of the material;
b) Manufacturer's name, initials or trade-mark, if any;
c) Volume of contents in litres;
d) Batch number; and
e) Manufacturing date.

4.2.3 *BIS Certification Marking*

The container may also be marked with the Standard Mark.

4.2.3.1 The use of Standard Mark is governed by provisions of the *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the license for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

5 SAMPLING

Representative samples of the material shall be drawn as prescribed in IS 1447 (Part 1).

Table 1 Requirements for Hexane, Food Grade
(*Clause* 3.2)

Sl No.	Characteristic	Requirement	Method of Test, Ref to	
			Annex	[P:] of IS 1448
(1)	(2)	(3)	(4)	(5)
i)	Density at 20°C,	0.660 - 0.687	—	[P : 32]
ii)	Refractive index, η_D^{20}	1.375 - 1.384	—	[P : 18]
iii)	Distillation:			
	(a) IBP, °C, *Min*	63		
	(b) Distilled between 64-70°C, percent volume, *Min*	95		
iv)	Non-volatile residue, g/100 ml, *Max*	0.0005	A	—
v)	Reaction of non-volatile residue	Neutral to methyl orange (*p*H indicator)	B	—
vi)	Sulphur content, mg/kg, *Max*	5 [2]	C or [3]	—
vii)	Lead, as Pb, mg/kg, *Max*	1	D	—
viii)	Benzene content, percent *v/v*, *Max*	0.05	E	—
ix)	Polycyclic aromatic hydrocarbons	To pass the test	F	—

[1] Test method is under preparation. Till such time, ASTM D 1218 shall be followed.

[2] Limit be 10 ppm till April 2003.

[3] Test method ASTM D 5453:1993/ASTM D 4045 (UV-Flourescence method can be used as alternate test method).

ANNEX A

[*Table* 1, *Sl No.* (iv)]

DETERMINATION OF NON-VOLATILE RESIDUE

A-1 SCOPE

This method prescribes the method for determination of non-volatile residue.

A-2 APPARATUS

Oven-thermostatically controlled at 105 ± 5°C.

A-3 PROCEDURE

Dry a 150-ml capacity glass evaporating dish in an oven at 105 ± 5°C and cool in a desiccator. Repeat until the weight is constant or within 0.1 mg of the last weighing. Pipette 100 ml of the sample at room temperature into the conditioned 150-ml glass evaporating dish; place on a steam bath and evaporate the sample to dryness. Dry the outside of the dish with a clean, lint-free cloth and heat in an oven at 105 ± 5°C for approximately 1 h. Cool in a desiccator and accurately weigh the contents and evaporating dish. Return the dish and contents to the oven for 15 to 30 min, cool and reweigh. Repeat, if necessary, until the weight is constant or with 0.1 mg of the last weighing. Preserve the residue for test as specified in **B-3**.

A-4 CALCULATION AND REPORTING

A-4.1 Calculate the non-volatile residue in the sample by means of the following equations:

$$\text{Non-volatile residue, g/100 ml} = \frac{A}{s} \times 100$$

where

A = mass in g of residue from evaporation, and

s = volume in ml of sample used.

A-4.2 Report the result to the third place of decimal

A-5 PRECISION

Results shall not differ from the mean by more than the following amounts:

	Repeatability	*Reproducibility*
Non-volatile residue, g/100 ml	0.000 5	0.001

ANNEX B

[*Table* 1, *Sl No.* (v)]

TEST FOR REACTION OF NON-VOLATILE RESIDUE

B-1 SCOPE

This method prescribes the test for determining the reaction of the non-volatile residue.

B-2 REAGENT

Methyl Orange Indicator — Dissolve 0.01 g of methyl orange in 100 ml of water.

B-3 PROCEDURE

B-3.1 To the conical flask containing the non-volatile residue as preserved in **A-3**, add 100 ml of distilled water and shake the contents thoroughly. Transfer the clean aqueous layer to a clean test tube by means of a pipette. Add one drop of methyl orange solution.

B-3.2 The residue shall be taken to have passed the test if no pink or red colour is formed.

ANNEX C

[*Table* 1, *Sl No.* (vi)]

DETERMINATION OF SULPHUR

(Adopted from UOP Method 357-80)

C-1 SCOPE

This method is used for the determination of low concentrations of sulphur, ranging from 0.1 to 200 ppm, in distillate stocks free of hydrogen sulphide. It may be applied to non-olefinic materials, such as straight run naphthas, hydrogen treated stocks, reformates and aromatic hydrocarbons. It is limited to samples containing not more than 2 percent olefins. Oxidized sulphur forms, such as sulphonic acids, are not determined quantitatively; therefore, this method must be applied with discretion to the analysis of stocks which have been treated with sulphuric acid.

C-2 OUTLINE OF METHOD

The sample is reacted with activated Raney nickel to convert organically-bound sulphur to nickel sulphide. Acid is then added to liberate the sulphur as hydrogen sulphide. The hydrogen sulphide is absorbed in a sodium hydroxide-acetone solution and titrated with standard mercuric acetate, using dithizone as the indicator.

C-3 APPARATUS

C-3.1 Apparatus — As shown in Fig. 1.

C-3.2 Balance — 'Torsion' or equivalent, 500 g capacity.

C-3.3 Burette — 10 ml.

C-3.4 Cylinders — Graduated 10 ml and 50 ml.

C-3.5 Filter Paper — Whatman No. 41.

C-3.6 Volumetric Flask — 1 000 ml, 2 000 ml.

C-3.7 Heating Mantle — For 100 ml flask.

C-3.8 Nitrogen Cylinder — Equipped with a pressure regulator. Arrange 2 lengths of rubber tubing to independently feed the acid reservoir and the flask respectively, as indicated on the drawing. Fit a pinchcock or a needle valve in each line to permit regulation of nitrogen flow.

C-3.9 Pipettes — Mohr, 1 ml.

C-3.10 Pipette — Volumetric, 1 ml, 5 ml and 10 ml.

C-3.11 Separatory Funnel — 125 ml.

C-3.12 Transformer — Variable.

C-4 REAGENTS AND MATERIALS

All reagents shall conform to the Indian Standards available, unless otherwise specified.

C-4.1 Water — *see* IS 1070.

C-4.2 Acetone — *see* IS 170.

C-4.3 Dithizone Solution — Dissolve 0.1 g of dithizone (diphenylthiocarbazone) in 100 ml of acetone. Prepare a new solution each week.

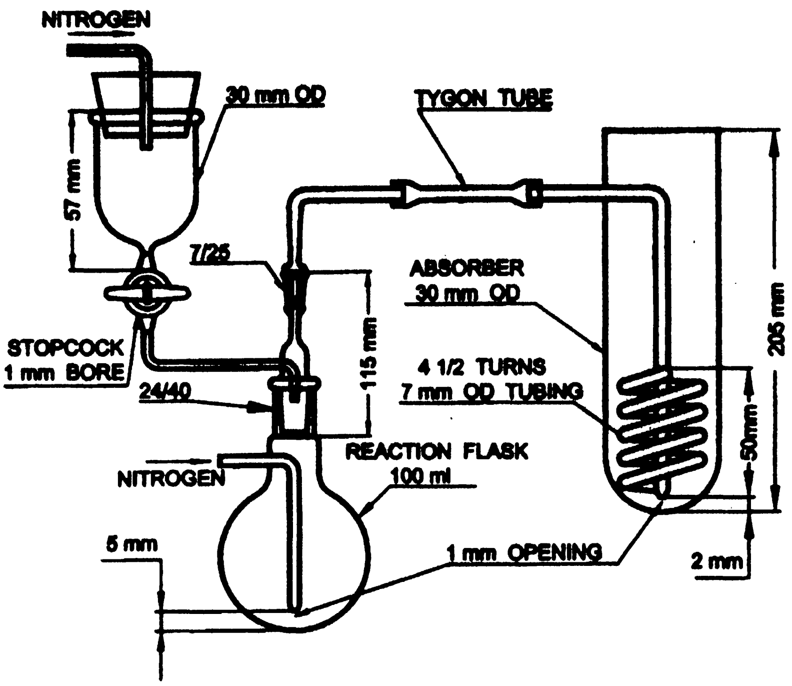


FIG. 1 APPARATUS FOR TRACE SULPHUR DETERMINATION

C-4.4 Dibutyl Disulphide — Also identified as butyl disulphide.

C-4.5 Hydrochloric Acid — Concentrated (*see* IS 265).

C-4.6 Hydrochloric Acid Solution — Dilute 1.5 volumes of concentrated hydrochloric acid with 1 volume of water.

C-4.7 *iso*-Octane — Pass over silica gel to remove sulphur compounds.

C-4.8 *iso*-Propyl Alcohol — *see* IS 2631.

C-4.9 Mercuric Acetate

C-4.9.1 Low-titre equivalent to 30 µgS/ml. Prepare by dissolving 0.41 g of mercuric oxide in 50 ml of aqueous solution containing 2 ml of acetic acid; dilute to 2 litre with water in a volumetric flask. Standardize this titrant by pipetting 5 ml of sulphur standard and following the analysis of a sample as described under **C-5**:

$$\text{Low-titre mercuric acetate solution, µgS/ml} = \frac{CD}{E}$$

where

C = sulphur standard, µgS/ml;

D = volume of sulphur standard used, ml; and

E = titration volume of low-titre mercuric acetate, ml.

C-4.9.2 *Mercuric Acetate* — High-titre, equivalent to 100 µgS/ml. Dissolve 1.35 g of mercuric oxide in 50 ml of aqueous solution containing 2 ml of acetic acid. When in solution, dilute to 2 litre with water in a volumetric flask. Standardize this titrant by pipetting 15 ml of the sulphur standard and follow the analysis of a sample as described under **C-5**:

$$\text{High-titre mercuric acetate solution, µgS/ml} = \frac{CD}{F}$$

where

F = titration volume of high-titre mercuric acetate, ml and C and D are defined in **C-4.9.1**.

NOTE — Granatelli found that approximately 95 percent recovery of the calculated added sulphur content was obtained when the complete analysis was carried out. Since the procedure is very precise, this is not an objectionable feature. However, the mercuric acetate solution must be standardized as specified above rather than by placing reliance on the molarity calculated from the weight of mercuric oxide used.

C-4.10 Mercuric Oxide — Yellow powder.

C-4.11 Nitrogen

C-4.12 Raney Nickel — Activated. Prepare the activated Raney nickel by placing 0.60 ± 0.1 g of nickel-aluminum (Raney catalyst) alloy in a 100 ml and add 10 ml of 2.5 M sodium hydroxide. Swirl the beaker gently in a hood until the vigorous evolution of hydrogen has ceased. Wash down any solids remaining on the sides of the beaker with a minimum of water. Allow the reaction to continue overnight in a covered vessel (such as a large desiccators minus desiccant). Store the activated Raney nickel in the excess sodium hydroxide, which is in the beaker and place it in a covered vessel until ready for use. It may be stored in the desiccator for as long as a week. This is enough activated Raney nickel for the analysis of one sample. The nickel prepared in this manner from 0.6 g of the alloy is enough to reduce at least 2 000 µg of sulphur.

C-4.13 Sodium Hydroxide — Approximately 2.5 M and 1 M.

C-4.14 Sulphur Standard — Place several gram of dibutyl disulphide into a dropping bottle. Weigh the bottle and contents to the nearest 0.1 mg. Add 60-100 mg of the disulphide to 1 litre of sulphur free *iso*-octane. Reweigh the dropping bottle to obtain the weight of the dibutyl disulphide removed. Dilute the contents of the 1litre flask to the mark with *iso*-octane and mix by inverting the flask several times. Calculate the titre in µgS/ml as:

$$\mu gS/ml\ (standard) = \frac{360.5A}{B}$$

where

A = mass of dibutyl disulphide, mg, and

B = volume of *iso*-octane, ml.

C-4.15 Wash Solution — Dissolve 100 g of cadmium chloride in 250 ml of water containing 10 ml of concentrated hydrochloric acid. Dilute to 1 litre with water. Chill to below 15°C and use immediately when called for as specified in **C-5**.

C-5 PROCEDURE

C-5.1 Thoroughly cleanse the reduction apparatus glassware with soap and water and then rinse at least two times with sulphur free acetone or alcohol. Dry in an oven at 110°C for one and a half hour or until dry. An excess of water inhibits the reduction of organic sulphur.

C-5.2 Test the sample for the presence of hydrogen sulphide by holding a piece of water moistened lead acetate paper in the vapour space of the sample container. If hydrogen sulphide is present the paper will darken or a silver sheen will appear. If hydrogen sulphide is not present proceed with the analysis. If hydrogen sulphide is present it must be removed as this method is applicable for organic sulphur only. The removal of hydrogen sulphide is obtained as specified in **C-5.3**.

C-5.3 Add up to 60 ml of sample to a 125 ml separatory funnel. Add 10 ml of cadmium chloride wash solution (freshly prepared) to the sample and shake 1 to 2 minutes. Allow the layers to separate and draw off the lower layer. If the test for hydrogen sulphide is strongly positive, continue additions of the wash solution until further additions do not produce turbidity. Wash three times, with 10 to 15 ml of water, discarding the water layers each time after shaking and letting the layers separate. Filter the sample rapidly with Whatman No. 41 filter paper and retain.

C-5.4 Decant the aqueous caustic solution from the beaker containing the activated Raney nickel just before using it. Wash the active nickel thoroughly 3 times with vigorous sprays of water. Allow the nickel to settle each time before the water is decanted, then rinse the water from the Raney nickel with 10 ml of *iso*-propyl alcohol and discard the rinsing.

C-5.5 Transfer the Raney nickel to the reaction flask with 10 ml of iso-propyl alcohol, rinsing down the walls of the flask with more alcohol in order to keep all of the Raney nickel submerged in the liquid. Weigh the flask containing the Raney nickel and *iso*-propyl alcohol to the nearest 0.05 g on a balance. Add the sample (for size of sample *see* Table 2) by pipette or graduate to the flask and reweigh the flask. Rely upon accurate pipetting for samples of 5 ml or less rather than weights obtained on the balance.

Table 2 Suggested Sample Size for Analysis
(*Clause* C-5.5)

Sl No.	Sulphur, ppm	Sample Size, ml	Concentration of Titrant, µgS/ml
(1)	(2)	(3)	(4)
i)	0-1	50	30
ii)	1-10	50	100
iii)	10-20	20	100
iv)	20-100	5	100
v)	100-200	2	100

C-5.6 Rinse the walls of the flask about 5 ml of *iso*-propyl alcohol to ensure contact of the entire sample with the Raney nickel. Place the flask in the heating mantle, lubricate the connecting head with stopcock grease and install it on the flask. Charge the absorber with 10 ml of 1 M sodium hydroxide, 10 ml of acetone, and 5 drops of dithizone solution. Assemble and connect the apparatus as indicated in the Fig. 1. Set the nitrogen pressure regulator for 2-5 psig and allow the nitrogen to flow through the reaction vessel and into the absorber at a rate of one bubble every 1s or 2s. Turn on the heater and allow the reactants to warm to a temperature at which no sample will be lost as a result of distillation during the sulphur reduction step (that is, use a temperature which is just short of inducing boiling). Allow the sulphur reduction to proceed for 30 min, agitating the contents of the flask several times during this period.

C-5.7 Charge the acid reservoir of the connecting head with 10 ml of 1.5:1 hydrochloric acid after the desulphurization is completed. Attach a nitrogen line loosely to the acid reservoir using a glass tube inserted in a neoprene stopper. Flush the air from the acid reservoir with a rapid flow of nitrogen and then reduce the rate of flow of the nitrogen and put the stopper firmly into place.

C-5.8 Add the acid drop wise to the reaction flask while maintaining the nitrogen flow through the reaction flask and into the absorber. Add just enough of the appropriate mercuric acetate titrant to the absorber liquid to change the colour of the solution from yellow to pink. After the vigorous reaction of the acid with the nickel has taken place, the remaining acid can be added rapidly. Maintain the nitrogen flow through the flask and reservoir. Increase the temperature to the point where the contents of the flask just barely reflux.

C-5.9 As the evolved hydrogen sulphide begins to be absorbed in the caustic solution, the colour of the solution will change from pink back to yellow. Titrate with the mercuric acetate solution until the absorber solution is definitely pink and remains that colour. Continue boiling the reaction flask contents for at least 30 min and then stop the nitrogen flow temporarily. Cool the reaction flask by applying a cold, wet cloth above the heating mantle, so that the absorber solution is sucked up in to the delivery tube. When the liquid approaches the side-arm, turn on the nitrogen to force the liquid back into the absorber. Repeat this operation several times, being careful not to allow any absorber solution to wet the horizontal portion of the delivery tube. If the absorber solution changes colour from pink to the yellow following this operation, titrate further the mercuric acetate to bring the solution to a permanent end point. Record the total volume of mercuric acetate titrant used. Obtain the net titration volume for the sample by subtracting the titration volume determined for the Raney nickel blank.

C-5.10 Determine a blank titration for each new batch of Raney nickel as specified in **C-5.10.1**.

C-5.10.1 Transfer the active nickel prepared from 0.6 g of the alloy to the reaction flask with 10 ml of *iso*-propyl alcohol. Rinse down the walls of the flask with about 5 ml of *iso*-propyl alcohol and attach the greased connecting-head. From this point on follow the procedure as described for the analysis of the sample with the exception that the sample is omitted. The total volume of the mercuric acetate required to reach the endpoint is the Raney nickel catalyst blank. The blank should amount to 0.1 to 0.2 ml of the 100 µgS titrant or 0.3-0.6 ml of the 30 µgS titrant. This amounts to 17-34 ppm sulphur in the Raney nickel alloy.

C-6 CALCULATION

$$\text{Sulphur, ppm} = \frac{AT}{M}$$

where

A = net volume of mercuric acetate solution used in titration, ml;

T = titre of the mercuric acetate solution, µgS/ml; and

M =mass of sample, g.

C-7 PRECISION

Duplicate results should be considered suspect if they differ by more than the following amounts:

Level of Sulphur, ppm	*Allowable Difference* ppm, *Sulphur*
1	0.3
10	0.6
50	2
250	6

The estimated standard deviation (σ_{SD}) based on the indicated replicates is as follows:

No. of Replicates	*Level*, ppm	σ_{SD}
5	1	0.08
5	10	0.16
4	50	0.71
4	100	2.6

C-8 TIME FOR ANALYSIS

The total elapsed time and man-hours are the same. The estimated man-hour requirement for one sample is 2.5 and 3.0 for 2 samples.

ANNEX D

[*Table* 1, *Sl No.* (vii)]

DETERMINATION OF LEAD

D-1 PRINCIPLE OF METHOD

The sample is treated with bromine, heated on a steam bath to decompose the alkyl lead and alkyl lead salts, and then extracted with dilute nitric acid. The *p*H of the aqueous extract is adjusted by means of a buffer and the lead is extracted with a chloroform solution of dithizone. The absorbance of the chloroform extract is measured and the lead content is determined from a previously prepared calibration curve.

D-2 APPARATUS

D-2.1 The glassware should be borosilicate and confirmed to be lead-free.

D-2.2 Spectrophotometer — Fitted with covered absorption cells having a 1 cm light path.

D-2.3 Shaking Machine (Optional) — Capable of approximately 250 rpm.

D-2.4 Separatory Funnel — Squibb-type 125 ml volume.

D-3 REAGENTS

D-3.0 Purity of Reagents — Reagent grade chemicals shall be used in all tests.

D-3.1 Purity of Water — Unless otherwise indicated, references to water shall be understood to mean distilled water or other water of equivalent purity.

D-3.2 Bromine Solution — Dilute 300 ml of bromine to 1 000 ml with chloroform. Filter through a sintered glass filter before using (Do in fume hood).

D-3.3 Buffer Solution — Dissolve 20 g of potassium cyanide (KCN), 6 g of ammonium citrate, and 6 g of sodium sulphite (Na2SO3) separately in water. Mix the solutions, add 150 ml of concentrated ammonium hydroxide (NH4OH, specific gravity 0.90) and dilute to 1 000 ml with water. This solution is stable for 3 months (in refrigerator).

D-3.4 Dithizone Solution — Dissolve 30 mg of dithizone in 1 000 ml of chloroform. This solution is stable for only 4 weeks.

D-3.5 Lead Standard Solution (1 ml = 0.005 mg Pb) Dissolve 1.598 5 g of lead nitrate [$Pb(NO_3)_2$] in 250 ml of water contained in a litre volumetric flask. Add 8 ml of concentrated HNO_3 (sp gr 1.42), dilute to volume with water, and mix thoroughly. For calibration purposes, pipette 5.0 ml of this solution into a 1-litre volumetric flask, add 8 ml of concentrated HNO_3 (sp gr 1.42), and dilute to volume with water.

D-3.6 Nitric Acid (sp gr 1.42) — Concentrated nitric acid (HNO_3).

D-3.7 Nitric Acid (8:992) — Mix 8 ml of concentrated HNO_3 (sp gr 1.42) with 992 ml of water.

D-4 CALIBRATION

D-4.1 Prepare a calibration curve using the lead solution (1 ml = 0.005 mg Pb) as follows:

> Pipette 0.0, 2.0, 5.0, 10.0 and 15.0 ml of the solution respectively into each of five separatory funnels and dilute each solution to 50 ml with HNO_3 (8:992). Treat these solutions as described in **D-5.3** and **D-5.4** using one not containing lead as the reagent blank.

D-4.2 Construct a calibration curve by plotting the absorbance of the solution against the mg of lead per 25 ml dithizone solution.

D-5 PROCEDURE

D-5.1 Pipette 50 g of the sample into a 250 ml beaker. Add bromine solution until the bromine colour persists for at least 2 min, then allow the beaker to stand for an additional 5 min. At the same time prepare a reagent blank by adding the same amount of bromine solution to 25.0 ml of HNO_3 (8:992) in a 250 ml beaker. Place the beaker on a steam bath and heat until the bromine colour disappears. Place the beakers on a hot plate and bring the solutions to a vigorous boil. Cool to room temperature and transfer each solution quantitatively, into separating funnels using 25.0 ml of HNO_3 (8:992). Shake the test sample for 2 min.

D-5.2 Drain the aqueous extract of the test sample into another separatory funnel. Repeat the extraction of the tests sample using 25.0 ml of HNO_3 (8:992). Drain the aqueous layer into the separatory funnel containing the initial extract. Transfer the reagent blank solution quantitatively to a 50 ml volumetric flask using 25.0 ml of HNO_3 (8:992). Dilute to the mark with water and drain the contents of the flask into a separatory funnel.

D-5.3 Add 120 ml of the buffer solution to both separatory funnels to adjust the *p*H to a point between

9.5 and 11.0. Pipette 25 ml of dithizone solution and shake for 2 min.

D-5.4 Drain and discard a small portion of the chloroform layer from the funnel to remove any water or lead that may have accumulated in the stem. Transfer a portion of each chloroform layer into separate absorption cells. Adjust the spectrophotometer to read zero absorbance for the reagent blank and then measure the absorbance of the sample with respect to the blank at a wavelength of 510 nm.

D-5.5 Convert the absorbance measurement to concentration of lead in milligrams of lead per 25 ml of dithizone solution by means of the previously prepared calibration curve.

D-6 CALCULATION

Calculate the lead content in mg per kg as follows:

$$\text{Lead, mg/kg} = \frac{100A}{50} = 20\,A$$

where

A = lead concentration, in mg/25 ml of dithizone solution corresponding to the measured absorbance.

ANNEX E

[*Table* 1, *Sl No.* (viii)]

TEST METHOD FOR BENZENE CONTENT

(Adopted from ASTM D-4367)

E-1 PRINCIPLE

An internal standard methyl ethyl ketone (MEK) is added to the test hexane which is then introduced in to a gas chromatograph equipped with two columns connected by flow diversion valve. The specimen passes first through a column packed with a non-polar phase, methyl silicone, which separates the non-polar hydrocarbons (column A). After octane has eluted, the flow through the non-polar column (column A) is reversed and diverted to a second column (column B) containing high polar phase. Components heavier than n-octane which have not eluted from column A are thus diverted to column B which separates the aromatic and non-aromatic compounds. The eluted components are detected by a flame ionization detector and recorded on a strip chart. The peak areas are measured and the concentration of each component is calculated by reference to the internal standard.

E-2 APPARATUS

E-2.1 Chromatograph — Any gas chromatograph (*see* Fig. 2) that has a backflush system, a flame ionization detector and that can be operated at the conditions given in Table 3 and suitable electronic recorder.

E-2.2 Columns — One 0.8 m long by 3.2 mm outside diameter stainless steel column packed with 10 percent methyl silicone coated on 60-80 mesh acid washed diatomaceous earth and one 4.6 m long by 3.2 mm outside diameter stainless steel column packed with 25 percent 1, 2, 3-Tris (2-cyanoethoxy) propane (TCEP) coated on 80-100 mesh acid washed diatomaceous earth. Join columns *A* and *B* as shown in Fig. 2 using an appropriate flow diverting valve. Adjust the helium gas flow through both columns to approximately 40 ml/mm with the flow valve in the forward flow position (*see* Fig. 3a). Set the valve in the backflush position and set the flow rate for column B to 40 ml/min (*see* Fig. 3b).

E-3 REAGENTS AND MATERIALS

Reagent grade chemicals having purity as given in **E-3.1** to **E-3.4** shall be used in all tests.

E-3.1 Methyl Ethyl Ketone (MEK) >99.9 percent.

E-3.2 Benzene >99 percent.

E-3.3 *iso*-Octane >99 percent.

E-3.4 *n*-Nonane >99 percent.

E-4 CALIBRATION

E-4.1 Determine time required for back flushing, before changing the valve which varies for each column system and must be determined experimentally as given in **E-4.1.1**.

E-4.1.1 Prepare a mixture of 5 volume percent *iso*-octane in n-nonane. With the system in the forward flow mode (*see* Fig. 2a) inject 1 ml of the *iso*-octane n-nonane mixture and allow both compounds to elute. Measure the time in seconds from the injection until all the *iso*-octane but none of the n-nonane has eluted. One-half of the measured time approximates the time to switch the backflush valve. Repeat the run, including

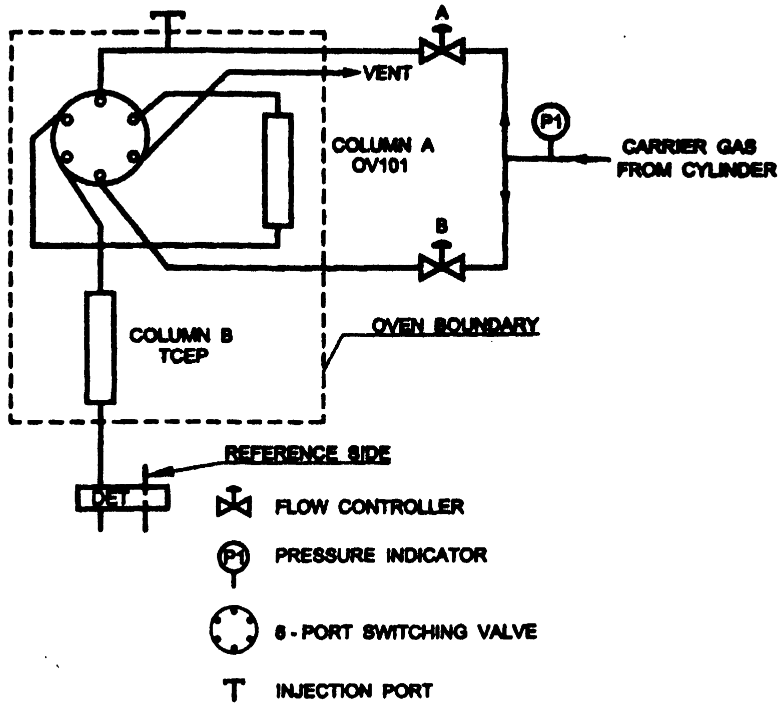
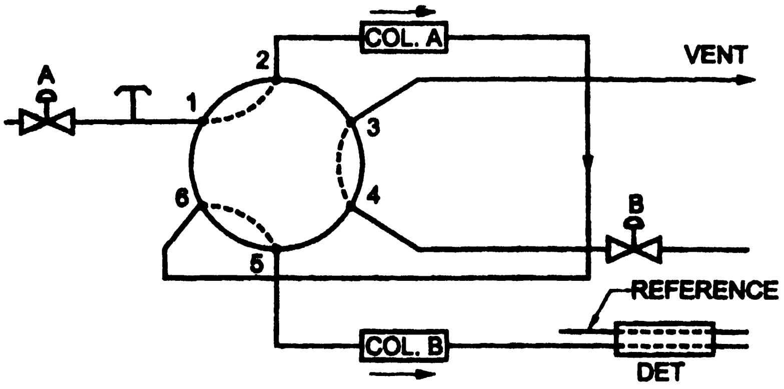
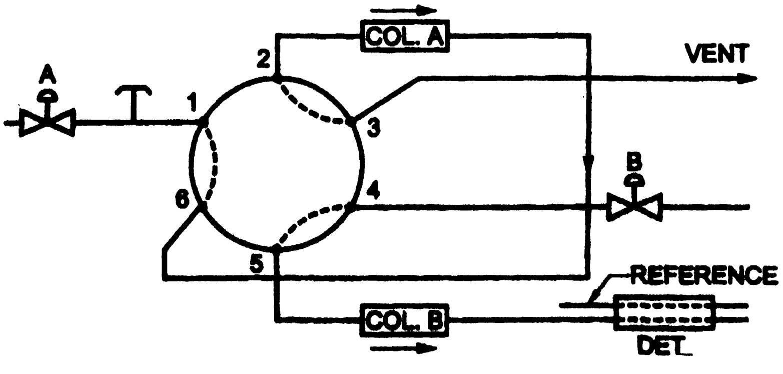


FIG. 2 PIPING AND INSTRUMENTATION



3a Forward Flow



3b Back Flush

FIG. 3 FLOW SWITCHING SYSTEM

the injection, but switching the system to the backflush mode at the determined backflush time. This should result in a chromatogram of *iso*-octane with little or no n-nonane evident. If necessary, make additional runs, adjusting the time to backflush until a chromatogram of all the *iso*-octane and little or none of the n-nonane is obtained. This established backflush time, including the actual valve operations, must be used in all subsequent calibrations and analysis.

E-4.2 Calibration and Standardization

E-4.2.1 *Standard Solutions*

Prepare seven standard solutions covering the range of 0 to 1 volume of benzene as prescribed in **E-4.2.1.1**.

E-4.2.1.1 For each standard, measure the volume of benzene listed below into a 100 ml volumetric flask. Dilute to volume with *iso*-octane, with all components and glassware at normal room temperature, and mix thoroughly:

Volume, Percent	ml
1	1
0.5	0.5
0.25	0.25
0.10	0.10
0.05	0.05
0.01	0.01
0.005	0.005

E-4.2.2 *Calibration Solutions*

Accurately measure 0.5 ml of MEK into a 100-ml volumetric flask, fill to the mark with the first standard solution and mix thoroughly. Repeat with each of the other standard solutions.

E-4.3 Chromatographic Analysis

Using the conditions given in Table 3, establish chromatograph for each of the calibration solutions after injecting approximately 1 μl.

E-4.4 Calibration

Measure the areas of the benzene and of MEK peaks. Calculate the ratio of the benzene peak area to the MEK peak area. Plot the concentration of benzene *versus* the ratio. The calibration must be done to ensure that the chromatographic system is operating properly and that the concentration of any one component has not exceeded the linear response range of any part of the system. The calibration plot should be linear. Determine the retention times for each component for future identification.

Table 3 Analytical Conditions
(*Clause* E- 4.3)

Injector temperature	150°C
Detector temperature	150°C
Column temperature (*iso*-thermal)	100°C
Carrier gas	Helium
Flow rate	30 ml/min
Sample size	1μl
Time to back flush	2 min (approximately)
Total cycle time	30 min

E-5 PROCEDURE

E-5.1 Test Solution — Accurately measure 0.5 ml of MEK in to a 100 ml volumetric flask. Fill to the mark with the material under test and mix well. Chromatograph a specimen from the test solution using the conditions given in Table 3. Identify on the chromatogram the benzene and the internal standard MEK peaks from the retention times of the standards. The order of elution is non-aromatic hydrocarbons, benzene, MEK and toluene when using the specified columns. Measure the areas under the benzene peak and under the MEK peak.

E-6 CALCULATION

Calculate the ratio of peak area of benzene to the peak area of MEK. Read from the calibration curve the volume percent of benzene corresponding to the calculated peak ratio. If the results are desired on a mass basis, convert to mass percent as follows:

$$\text{Benzene, percent by mass} = (V/D) \times 0.884\,4$$

where

V = benzene volume percent, and

D = relative density of sample at 15.6°C/15.6°C.

Report the benzene content in volume or mass percent the nearest 0.005 percent.

E-6.1 Repeatability

Two results each the mean of duplicates should not differ more than 0.027 percent absolute.

E-6.2 Reproducibility

Two results each the mean of duplicates should not differ more than 0.063 percent absolute.

ANNEX F

[*Table* 1, *Sl No.* (ix)]

DETERMINATION OF POLYCYCLIC AROMATIC HYDROCARBONS

F-1 SCOPE

This method prescribes the test for determining the polycyclic aromatic hydrocarbons in food grade hexane sample.

F-2 GENERAL INSTRUCTIONS

All glassware should be scrupulously cleaned to remove all organic matter such as oil, grease, detergent residue, etc. Examine all glassware, including stopper and stopcocks, under ultraviolet light to detect any residual fluorescent contamination. As a precautionary measure, it is recommended practice to rinse all glassware with purified *iso*-octane immediately before use. No grease is to be used on stopcocks or joints. Because some of the polynuclear hydrocarbons sought in this test are very susceptible to photo-oxidation, the entire procedure is to be carried out under subdued light.

F-3 APPARATUS

F-3.1 Separatory Funnel — 125 ml capacity equipped with tetrafluoroethylene (PTFE) polymer stopcocks.

F-3.2 Spectrophotometric Cells — Fused quartz cells, optical pathlength in the range of 5.0 ± 0.005 cm; also for checking spectrophotometer performance only, optical pathlength in the range 1.0 ± 0.005 cm. With distilled water in the cells, determine any absorbance difference.

F-3.3 Spectrophotometer — Spectral range 200 - 450 nm with spectral slit width of 2 nm or less; under instrument operating conditions for these absorbance measurements, the spectrometer shall also meet the following performance requirements:

Absorbance repeatability : ± 0.01 at 0.4 absorbance

Absorbance accuracy : ± 0.05 at 0.4 absorbance

Wavelength repeatability : ± 0.2 nm

Wavelength accuracy : ± 1.0 nm

F-4 REAGENTS

F-4.1 *iso*-Octane (2, 2, 4-trimethyl Pentane) — UV spectroscopic grade. If necessary, *iso*-octane may be purified by passing through a column of activated silica gel (Grade 1) or by distillation.

F-4.2 Dimethyl Sulphoxide (DMSO) — UV spectroscopic grade.

F-4.3 n-Hexane — UV spectroscopic grade.

F-4.4 Naphthalene — Analytical grade naphthalene.

F-5 PROCEDURE

F-5.1 Preparation of Standard (Reference) Solution of Naphthalene and Measurement of Absorbance

Prepare a standard (reference) solution of naphthalene in *iso*-octane containing 7.0 mg of naphthalene per litre. Measure the absorbance of the standard (reference) solution at 275 nm using *iso*-octane as blank.

F-5.2 Determination of UV Absorbance of the Test Solution

Add 25 ml of n-hexane (UV spectroscopic grade) to a 125 ml separating funnel. Add 5 ml of dimethyl sulphoxide (DMSO). Shake vigorously for 1 min and allow to stand until two clear layers are formed. Collect the clear lower layer and use as blank to measure the absorbance of the test solution.

a) Take two 125 ml separating funnels. Transfer 25 ml of sample (food grade hexane) to the first 125 ml separating funnel and add 25 ml of n-hexane (UV spectroscopic grade). Mix and add 5 ml of dimethyl sulphoxide (DMSO). Shake vigorously for 1 min and allow to stand until two clear layers are formed.

b) Transfer the lower layer to a second separating funnel, add 2 ml of n-hexane (UV spectroscopic grade) and shake the mixture vigorously. Allow to stand until two clear layers are formed. Separate the lower layer and measure the absorbance over the range of 260 nm to 420 nm against the blank.

F-5.3 At any wavelength in the range 260 nm to 420 nm the absorbance of the test solution should not exceed one-third that of the reference (standard) solution at 275 nm.

ANNEX G
(*Foreword*)

COMMITTEE COMPOSITION
Petroleum Products Sectional Committee, PCD 3

Organization	*Representative(s)*
Indian Additives Ltd, Chennai	Shri R. A. Rao (***Chairman***) Shri G. S. Ramachandran (*Alternate*)
Ashok Leyland Ltd, Chennai	Shri M. Nirmal Kumar Shri S. K. Raju (*Alternate*)
Association of State Road Transport Undertaking (ASRTU), Pune	Shri A. S. Lakra
Automotive Research Association of India, Pune	Shri M. K. Chaudhari Shri A. R. Arankalle (*Alternate*)
Bharat Petroleum Corporation Ltd, Mumbai	Dr Y. P. Rao Shri R. Ramachandran (*Alternate*)
Bongaigaon Refinery & Petrochemicals Ltd, Dhaligaon (Assam)	Shri D. K. Phuran Shri Sagar Mathews (*Alternate*)
Centre For High Technology, New Delhi	Shri S. K. Singh Shri B. D. Ghosh (*Alternate*)
Central Institute of Road Transport (Training & Research), Pune	Shri B. K. Chapra Shri N. R. Kachare (*Alternate*)
Central Revenue Control Laboratory, New Delhi	Chief Chemist Deputy Chief Chemist (*Alternate*)
Chennai Petroleum Corporation Ltd, Chennai	Shri K. Raman Shri N. G. Subramanian (*Alternate*)
Chief Controller of Explosives, Nagpur	Dr A. S. Ghosal Shri R. S. Bhalekar (*Alternate*)
Cochin Refineries Ltd, Eranakulam	Shri C. I. Roy Shri K. P. Subramanian (*Alternate*)
Controllerate of Quality Assurance Petroleum Products, Ministry of Defence, DGQA, New Delhi	Shri S. K. Srivastava Shri Vijaya Kumar (*Alternate*)
Defence Materials And Stores Research & Development Establishment, Kanpur	Dr P. S. Venkataramani Dr A. K. Mehrotra (*Alternate*)
Directorate General Aeronautical Quality Assurance, Ministry of Defence, New Delhi	Director Shri G. S. Bhatnagar (*Alternate*)
Directorate General of Civil Aviation, New Delhi	Shri B. K. Joshi Shri S. S. Muzumdar (*Alternate*)
Directorate of Maintenance Plans & Inspection (Air HQ), Ministry of Defence, New Delhi	Joint Director (QAS) Deputy Director (QAS) (*Alternate*)
Escorts Ltd, Faridabad	Shri Shive D. Khanna Shri H. C. Bahuguna (*Alternate*)
Exxon Chemical, Mumbai	Shri S. N. Sinha Shri M. S. Shesha Sayee (*Alternate*)
Hero Honda Motors Ltd, Dist Mohinder Garh, Haryana	Shri S. B. Gupta Shri P. K. Salhotra (*Alternate*)
Hindustan Petroleum Corporation Ltd, Visakhapattanam	Shri B. Saranathan Shri P. Seeta Rama Rao (*Alternate*)
Hindustan Petroleum Corporation Ltd, Mumbai	Shri M. K. Daxini
Ind-Auto Limited, Mumbai	Shri P. N. Burgulas Shri Y. J. Khanvekar (*Alternate*)
IBP Company Ltd, Kolkata	Shri A. G. Biswas Shri Vinod Moudgil (*Alternate*)
Indian Institute of Petroleum, Dehra Dun	Shri Sudhir Singhal Dr M. O. Garg (*Alternate*)
Indian Institute of Technology Kanpur	Dr B. P. Pundir
Indian Oil Corporation Ltd (Marketing Division), Mumbai	Shri P. D. Bahukhandi Shri H. K. Sayal (*Alternate*)
Indian Oil Corporation Ltd (R & P Division), New Delhi	Shri K. K. Acharya Shri K. Balachandran (*Alternate*)
Indian Oil Corporation Ltd (Research & Development Centre), Faridabad	Shri N. R. Raje Shri B. S. Rawat (*Alternate*)
LML Ltd, Kanpur	Shri Dinesh Kumar
Lubrizol India Ltd, Mumbai	Shri M. R. Khumbhani Shri V. P. Wagh (*Alternate*)

(*Continued on page*

(*Continued from page* 12)

Organization	*Representative(s)*
Mangalore Refinery & Petrochemical Ltd, Mangalore	Shri R. Rajamani Shri S. Ramesh (*Alternate*)
Maruti Udyog Ltd, Gurgaon, Haryana	Dr K. Kumar Manager Engineering (*Alternate*)
Ministry of & Environment Forest, New Delhi	Dr S. V. Reddy Dr B. Sengupta (*Alternate*)
Ministry of Industry, New Delhi	Shri B. Bhanot Shri S. K. Jain (*Alternate*)
Ministry of Petroleum and Natural Gas, New Delhi	Adviser *(Refineries)* Officer on Special Duty, Technical (*Alternate*)
Ministry of Surface Transport, New Delhi	Shri S. N. Pattnayak Shri Virendra Singh (*Alternate*)
National Test House, Kolkata	Dr Y. C. Nijhan Shri A. N. Bansood (*Alternate*)
Naval Headquarters, New Delhi	Director of Systems Engg. Assistant Director of Systems Engineering (*Alternate*)
Oil Industry Safety Directorate, New Delhi	Shri S. C. Gupta Shri S. N. Mathur (*Alternate*)
Petroleum Conservation Research Association, New Delhi	Shri U. P. Singh Shri Paramjit Singh (*Alternate*)
Research, Designs and Standards Organization (RDSO), Lucknow	Deputy Director (AROCM III) Assistant Research Officer (CM-3) (*Alternate*)
Reliance Petroleum Limited, Mumbai	Shri N. S. Murthy Shri R. V. Desai (*Alternate*)
Society of Indian Automobile Manufacturers, New Delhi	Ms Pamella Tikku Shri K. K. Gandhi (*Alternate*)
Tata Engineering & Locomotive Co Ltd, Jamashedpur	Shri V. Laxminarayan Shri T. Seturamalingam (*Alternate*)
BIS Directorate General	Shri Anjan Kar, Director & Head (PCD) [Representing Director General (*Ex-officio*)]

Member Secretary
Shri R. P. Mishra
Joint Director (PCD), BIS

Petroleum Solvents Subcommittee, PCD 3:7

Indian Oil Corporation Ltd (R & D Centre), Faridabad	Shri B. S. Rawat (***Convener***) Shri S. C. Mehta (*Alternate*)
Bharat Petroleum Corporation Ltd, Mumbai	Shri S. V. Kharat Shri R. Armugam (*Alternate*)
Bongaigaon Refinery & Petro-Chemical Ltd, Dhaligaon	Shri C. P. Bezboruah Shri S. Ghosh (*Alternate*)
Central Committee for Food Standards, Directorate General of Health, New Delhi	Shri Prem K. Gupta Shrimati Debi Mukherjee (*Alternate*)
Chennai Petroleum Corporation Ltd, Chennai	Shri K. Raman Shri M. S. Shayamsunder (*Alternate*)
Cochin Refinery Ltd, Amabalamugal	Shri V. H. Muhammed Shri V. Paily (*Alternate*)
Directorate of Vanaspati Vegetable Oils & Fats, New Delhi	Dr M. K. Kundu Shri V. P. Awasthy (*Alternate*)
Hindustan Petroleum Corporation Ltd, Mumbai	Shri M. K. Daxini
Indian Oil Corporation Ltd (Marketing), Mumbai	Shri A. M. Nagar Shri S. K. Gupta (*Alternate*)
IOC (Assam Oil Division), Assam	Shri I. P. Baruah Shri A. K. Chakravorty (*Alternate*)
Indian Institute Petroleum, Dehra Dun	Dr M. K. Khanna Shri Srikant Nanoti (*Alternate*)
Indian Oil Corporation Ltd (R & P Division), New Delhi	Shri Ananda Rao Shri U. K. Roy (*Alternate*)
Indian Paint Association, Kolkata	Dr A. B. Gersappa
National Organic Chemical Industrial Ltd, Thane	Shri B. V. Bapat Shri M. B. Surve (*Alternate*)
The Solvent Extractors' Association of India, Mumbai	Shri Bharat Mani Shri S. R. Lunkad (*Alternate*)

Bureau of Indian Standards

BIS is a statutory institution established under the *Bureau of Indian Standards Act*, 1986 to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Copyright

BIS has the copyright of all its publications. No part of these publications may be reprodued in any form without the prior permission in writing from BIS. This does not preclude the free use, in the course of implementing the standard, of necessary details, such as symbols and sizes, type or grade designations. Enquiries relating to copyright may be addressed to the Director (Publications), BIS.

Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards: Monthly Additions'.

This Indian Standard has been developed from Doc : No. PCD 3 (1638).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters :

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110 002
Telephones : 323 01 31, 323 33 75, 323 94 02

Telegrams : Manaksanstha
(Common to all offices)

Regional Offices :		Telephone
Central	: Manak Bhavan, 9 Bahadur Shah Zafar Marg NEW DELHI 110 002	323 76 17 323 38 41
Eastern	: 1/14 C.I.T. Scheme VI M, V. I. P. Road, Kankurgachi KOLKATA 700 054	337 84 99, 337 85 61 337 86 26, 337 91 20
Northern	: SCO 335-336, Sector 34-A, CHANDIGARH 160 022	60 38 43 60 20 25
Southern	: C.I.T. Campus, IV Cross Road, CHENNAI 600 113	254 12 16, 254 14 42 254 25 19, 254 13 15
Western	: Manakalaya, E9 MIDC, Marol, Andheri (East) MUMBAI 400 093	832 92 95, 832 78 58 832 78 91, 832 78 92

Branches : AHMEDABAD. BANGALORE. BHOPAL. BHUBANESHWAR. COIMBATORE. FARIDABAD. GHAZIABAD. GUWAHATI. HYDERABAD. JAIPUR. KANPUR. LUCKNOW. NAGPUR. NALAGARH. PATNA. PUNE. RAJKOT. THIRUVANANTHAPURAM. VISAKHAPATNAM.

Printed by Dee Kay Printers.